



## **Microfabricated Chemical Sensors for Aerospace Fire Detection Applications**

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# Instrumentation & Control Technology Division

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## OUTLINE

- INTRODUCTION
- MICROFABRICATED GAS SENSORS
- SENSOR DEVELOPMENT

HYDROGEN

CARBON MONOXIDE AND NITROGEN OXIDES

CARBON DIOXIDE

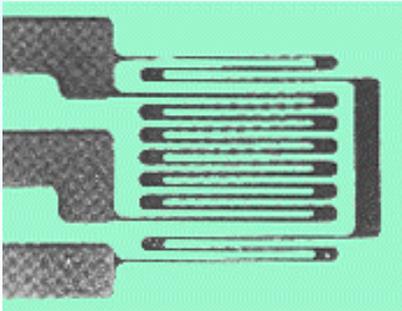
OXYGEN

HYDROCARBONS

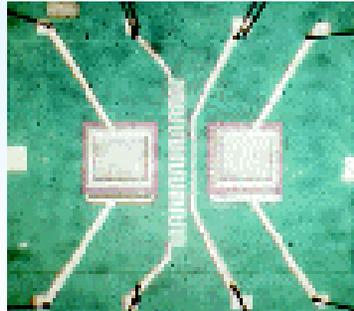
- HIGH TEMPERATURE ELECTRONIC NOSE
- FIRE DETECTION NEEDS AND APPROACH
- SUMMARY AND FUTURE PLANS

## SENSORS & ELECTRONICS TECHNOLOGY BRANCH

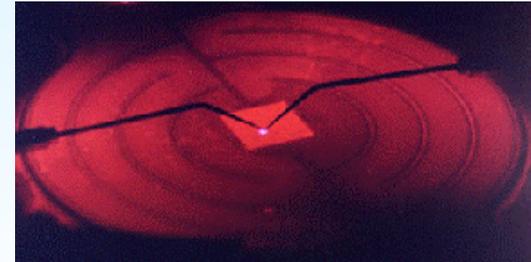
### SCOPE OF WORK



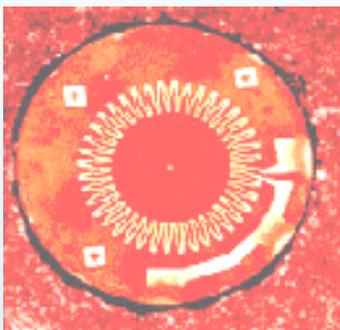
**STRAIN GAGES**



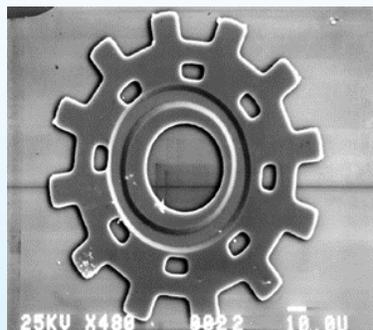
**CHEMICAL SENSORS**



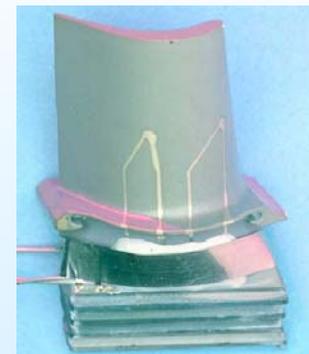
**SILICON CARBIDE HIGH TEMPERATURE ELECTRONICS**



**HEAT FLUX GAGES**



**MICROELECTROMECHANICAL SYSTEMS (MEMS)**



**TEMPERATURE SENSORS**



# Instrumentation & Control Technology Division

## MICROSYSTEMS

**MEMS:** Micro ElectroMechanical Systems (Sensors, Electronics, and Actuators)

**MICROSYSTEMS:** Sensors and devices created using microfabrication and micromachining techniques (Silicon electronics based processing)

**BENEFITS:** Reduced Size, Weight, and Power Consumption of Components With Significantly Increase Capabilities

### APPLICATIONS

Smart Engine Control (e.g. noise, stall)  
Emissions Reduction  
Engine Tests  
Structure and Material Development

Safety/Ice Detection  
Health Monitoring  
CFD Code Validation  
Fuel Delivery (Atomizers)

### ACTIVITIES

High Temperature Pressure Sensor  
Emission Sensors  
Heat Flux Sensor Array

Shear Stress Sensor  
Thin Film Sensors  
High Temperature Electronics

### TECHNICAL CHALLENGES

Packaging  
Material Stability, Compatibility

System Integration  
Corresponding Actuator Systems



# **Instrumentation & Control Technology Division**

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## **MICROFABRICATION AND MICROMACHINING TECHNOLOGY**

### **MEMS-BASED TECHNOLOGY**

- **MICROELECTRONIC FABRICATION TECHNIQUES APPLIED TO CHEMICAL SENSOR DEVELOPMENT**

  - PHOTOLITHOGRAPHIC REDUCTION**

  - THICK AND THIN FILM METALLIZATION**

  - SPUTTERING AND ELECTRON-BEAM DEPOSITION**

  - BATCH PROCESSING/MINIMAL COST**

  - PRECISE CONTROL OF STRUCTURE**

  - MINIMAL SIZE, WEIGHT, AND POWER CONSUMPTION**

- **MICROMACHINING FOR THREE DIMENSIONAL STRUCTURES**

  - ETCHING AND SACRIFICIAL LAYER METHODS (MINIMIZE HEAT CONSUMPTION)**

  - FABRICATE COMPLEX SHAPES**

  - TAILOR SENSOR STRUCTURE**



## MICROFABRICATED GAS SENSORS

- COLLABORATIVE EFFORT BETWEEN NASA GRC AND CASE WESTERN RESERVE UNIVERSITY
- SENSOR DEVELOPMENT RESULTING FROM:
  - IMPROVEMENTS IN MICROFABRICATION AND MICROMACHINING TECHNOLOGY
  - DEVELOPMENT OF SiC-BASED SEMICONDUCTOR TECHNOLOGY
- GAS DETECTION IN:
  - HARSH ENVIRONMENTS
  - APPLICATIONS BEYOND CAPABILITIES OF COMMERCIAL SENSORS

TECHNOLOGY DEVELOPS PLATFORMS FOR A VARIETY OF MEASUREMENTS

SCHOTTKY DIODE  
RESISTANCE BASED  
ELECTROCHEMICAL

- TARGET DETECTION OF GASES OF FUNDAMENTAL INTEREST

HYDROCARBONS ( $C_xH_y$ )  
NITROGEN OXIDES ( $NO_x$ ) AND CARBON MONOXIDE (CO)  
OXYGEN ( $O_2$ )  
CARBON DIOXIDE ( $CO_2$ )  
HYDROGEN ( $H_2$ )



## CHEMICAL SENSOR APPLICATION DEVELOPMENT AREAS

### SAFETY

#### FIRE DETECTION

DETECTION OF FIRE PRECURSORS (E.G. CO AND CO<sub>2</sub>) IN CARGOBAY APPLICATIONS TO SUPPLEMENT EXISTING TECHNOLOGY. CHEMICAL SIGNATURE IN THE PRESENCE OF A NUMBER OF INTERFERING GASES. COMPLEMENT EXISTING SMOKE DETECTION SYSTEMS.

#### LEAK DETECTION

DETECTION OF FUEL AND OXYGEN LEAKS FOR SPACE TRANSPORTATION APPLICATIONS SUCH AS SPACE SHUTTLE, X-33 AND BANTAM. WIDE RANGE DETECTION IN INERT ENVIRONMENTS AND POSSIBLY CRYOGENIC CONDITIONS.

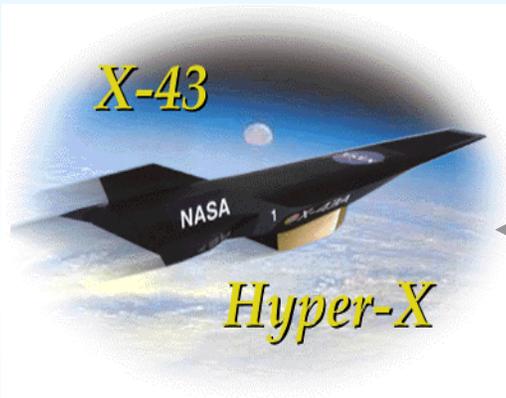
### EMISSIONS

DETECTION OF HYDROCARBONS, NO<sub>x</sub>, CO, ETC. FOR HEALTH MONITORING AND ACTIVE COMBUSTION CONTROL APPLICATIONS. SENSITIVE DETECTION IN HIGH TEMPERATURE HARSH ENVIRONMENTS IN THE PRESENCE OF A NUMBER OF INTERFERING GASES.

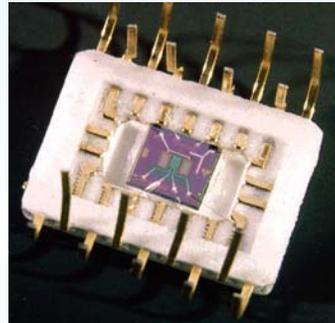
## *HYDROGEN LEAK SENSOR TECHNOLOGY*

**1995 R&D 100 AWARD WINNER**

- ◆ MICROFABRICATED USING MEMS-BASED TECHNOLOGY FOR MINIMAL SIZE, WEIGHT AND POWER CONSUMPTION
- ◆ HIGHLY SENSITIVE IN INERT OR OXYGEN-BEARING ENVIRONMENTS, WIDE CONCENTRATION RANGE DETECTION
- ◆ INTEGRATED WITH SMART ELECTRONICS FOR SIGNAL PROCESSING AND TEMPERATURE CONTROL



SYSTEM DELIVERED FOR HYPER X FLIGHT PLANNED FOR SUMMER 00



DEMONSTRATED ON STS-95 and STS-96 SHUTTLE MISSIONS



20 SENSOR SYSTEM DELIVERED FOR X-33 SAFETY SYSTEM

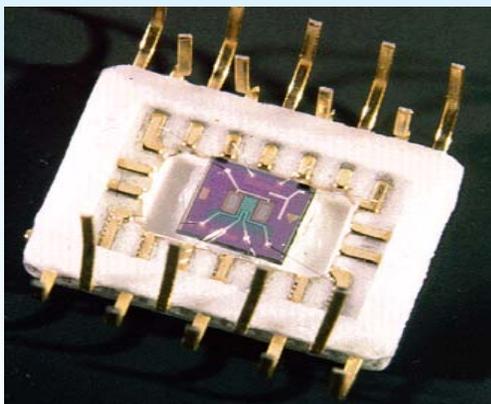


AUTOMATED HYDROGEN LEAK DETECTION SYSTEM ON NATURAL GAS POWERED CROWN VICTORIA ASSEMBLY LINE



CHOSEN FOR INCLUSION ON ISS WATER PROCESSING O2 GENERATOR.

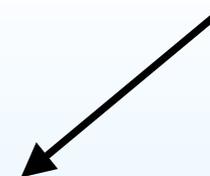
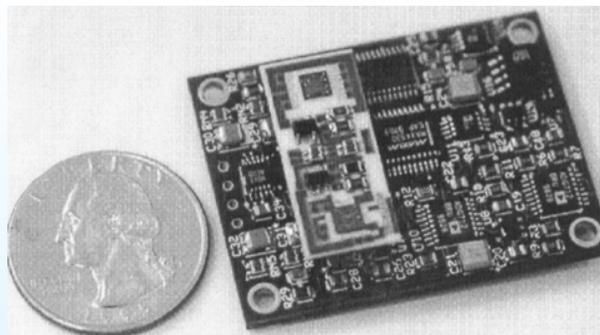
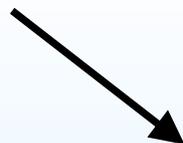
## MINIATURIZED SMART LEAK SENSOR SYSTEM



**MICROFABRICATED  
HYDROGEN SENSOR**



**HYDROGEN SENSORS ON  
SPACE SHUTTLE**



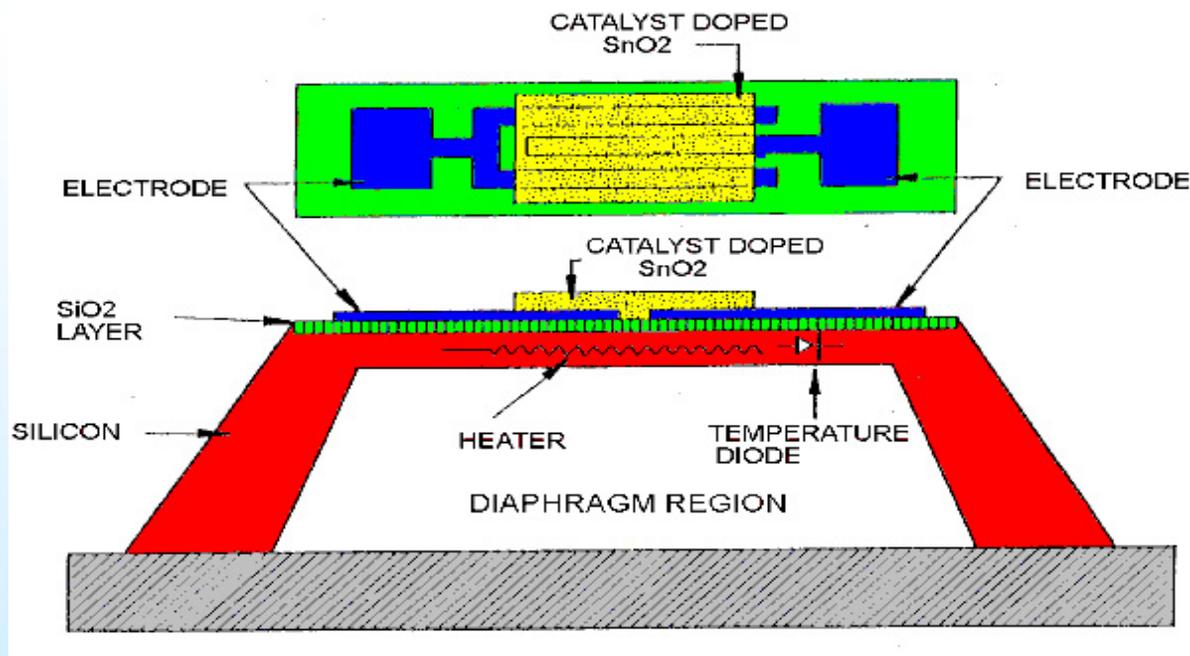
**PROTOTYPE HYDROGEN/OXYGEN SENSOR SYSTEM WITH ELECTRONICS**



**DEMONSTRATE STAND-ALONE SMART LEAK DETECTION SYSTEM  
WITH A SURFACE AREA THE SIZE OF POSTAGE STAMP**

## *MICROFABRICATED TIN OXIDE BASED NO<sub>x</sub> AND CO SENSOR TECHNOLOGY*

- MICROFABRICATED FOR MINIMAL SIZE, WEIGHT AND POWER CONSUMPTION
- MICROMACHINED TO MINIMIZE POWER CONSUMPTION AND IMPROVE RESPONSE TIME
- TEMPERATURE DETECTOR AND HEATER INCORPORATED INTO SENSOR STRUCTURE
- NANOFABRICATION OF TIN-OXIDE TO INCREASE SENSOR STABILITY



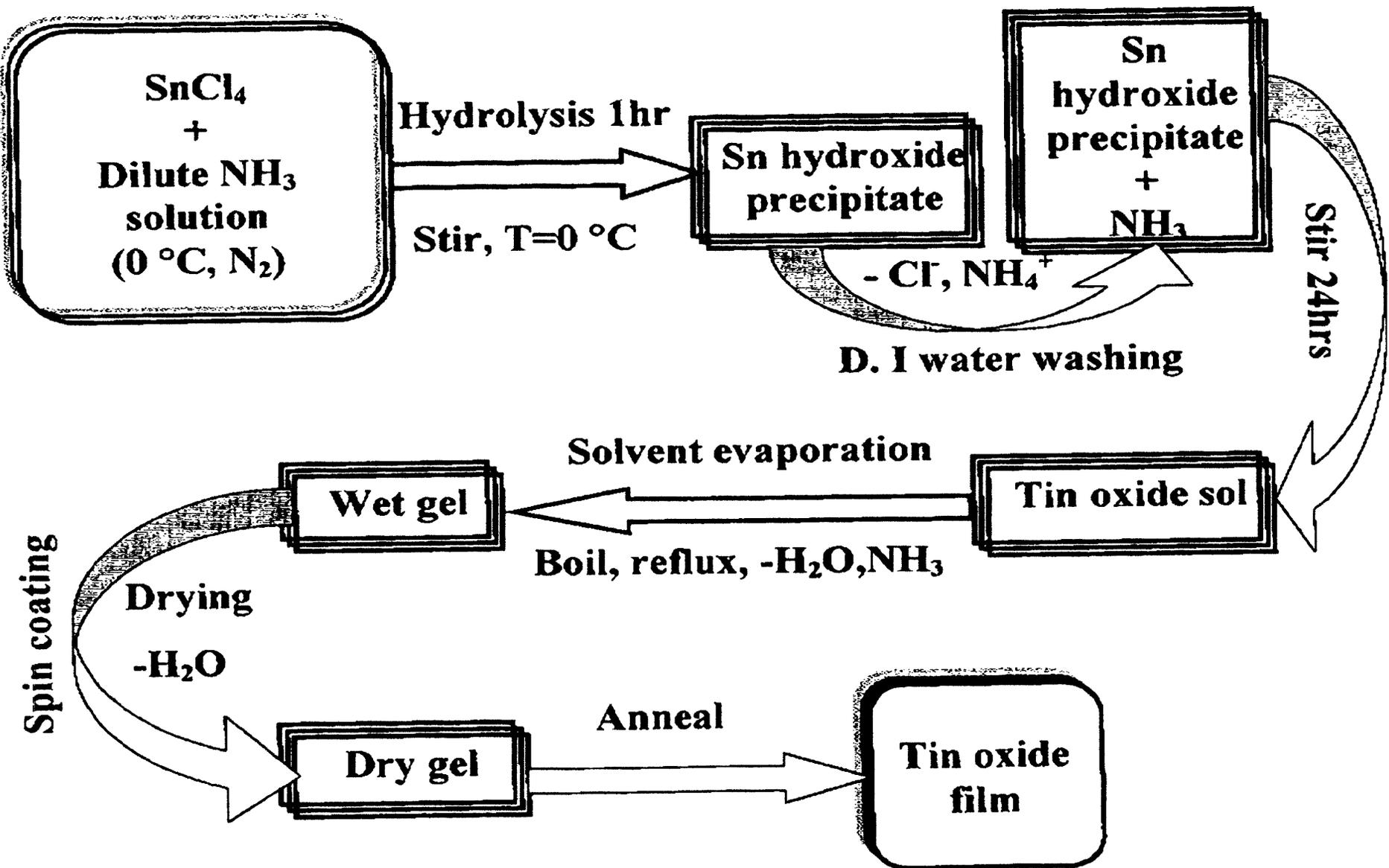
**STRUCTURE OF A MICROFABRICATED TIN-OXIDE SENSOR**

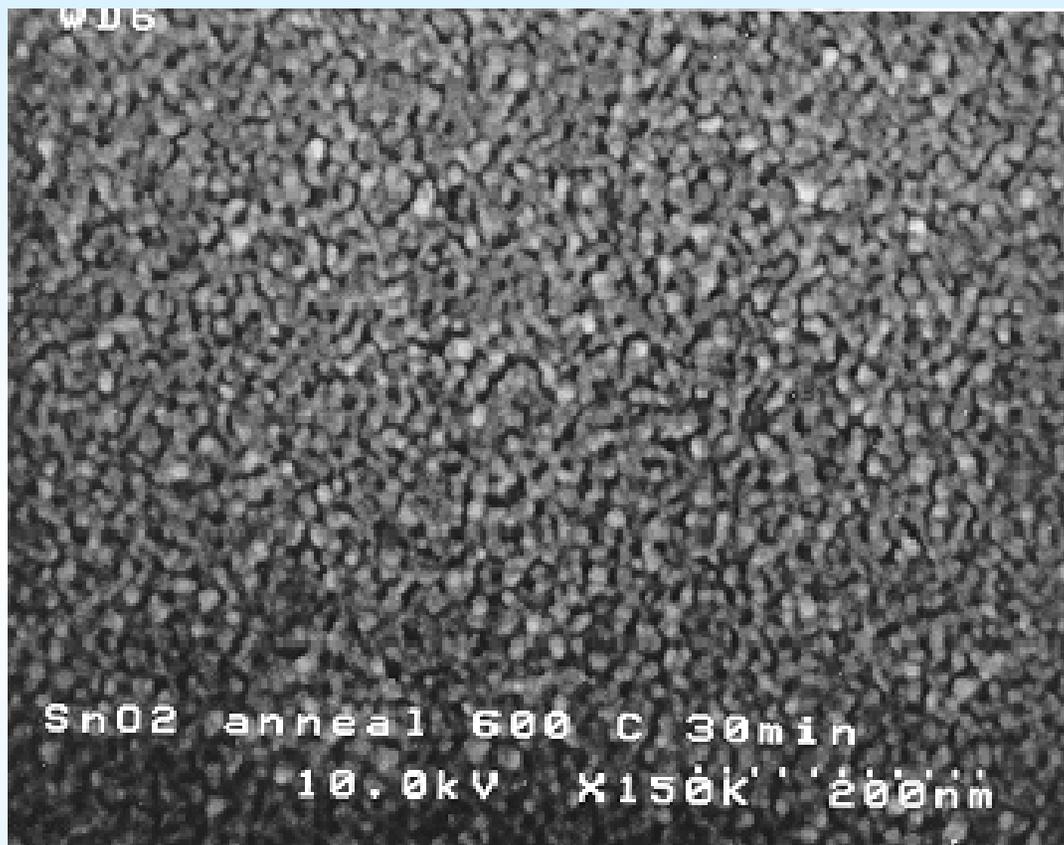
## PROPERTIES OF NANOCRYSTALLINE $\text{SnO}_2$ THIN FILM FROM SOL-GEL PROCESS

- SMALL PARTICLE SIZE
- HIGH POROSITY
- LARGE SURFACE AREA
- HOMOGENEOUS CHEMICAL AND PHYSICAL STRUCTURE
- DIFFERENT ANNEALING MECHANISMS/RATES

## ADVANTAGES FOR SENSOR APPLICATIONS

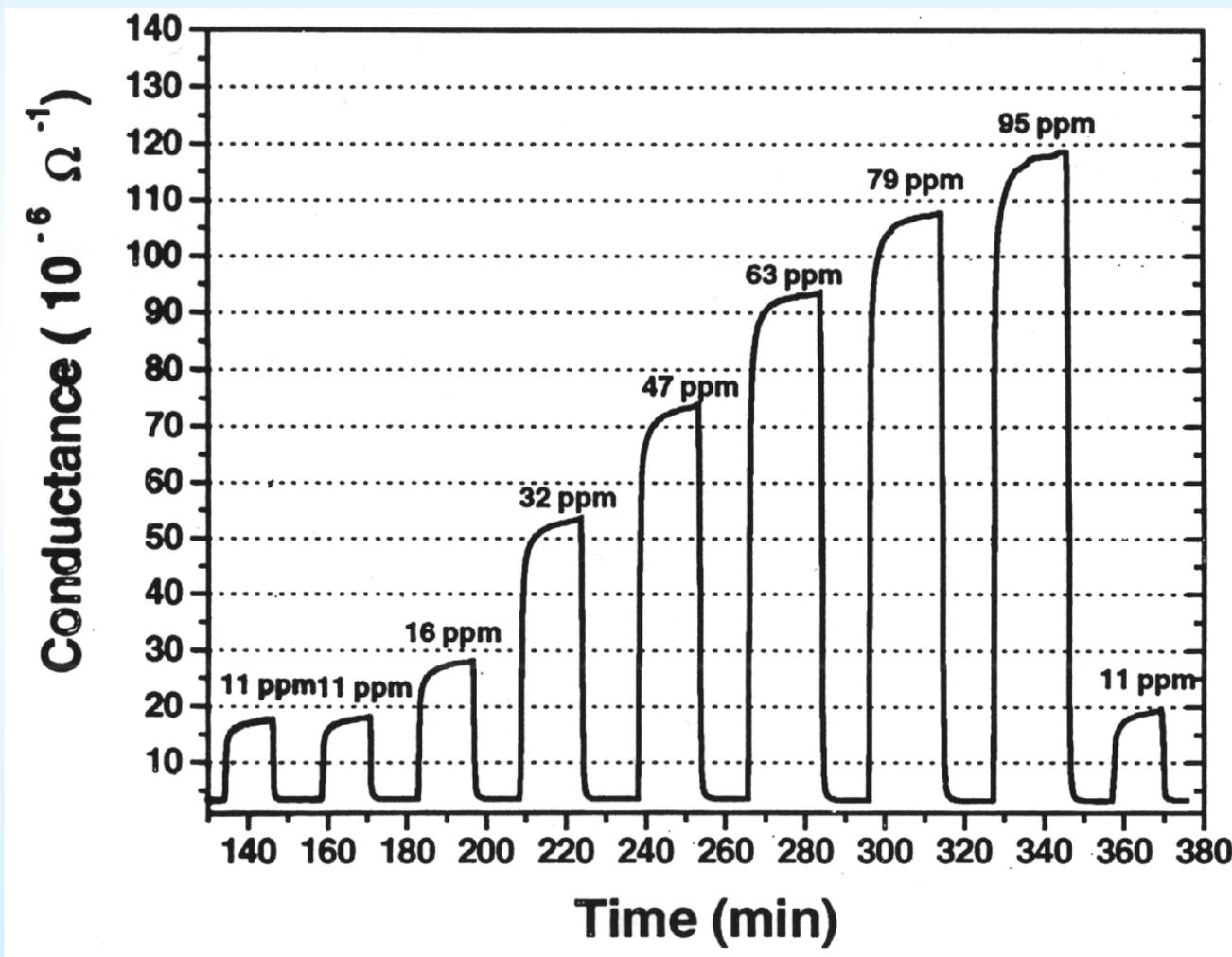
- HIGH SENSITIVITY
- FAST RESPONSE
- STABLE OPERATION
- LOWER TEMPERATURE OPERATION



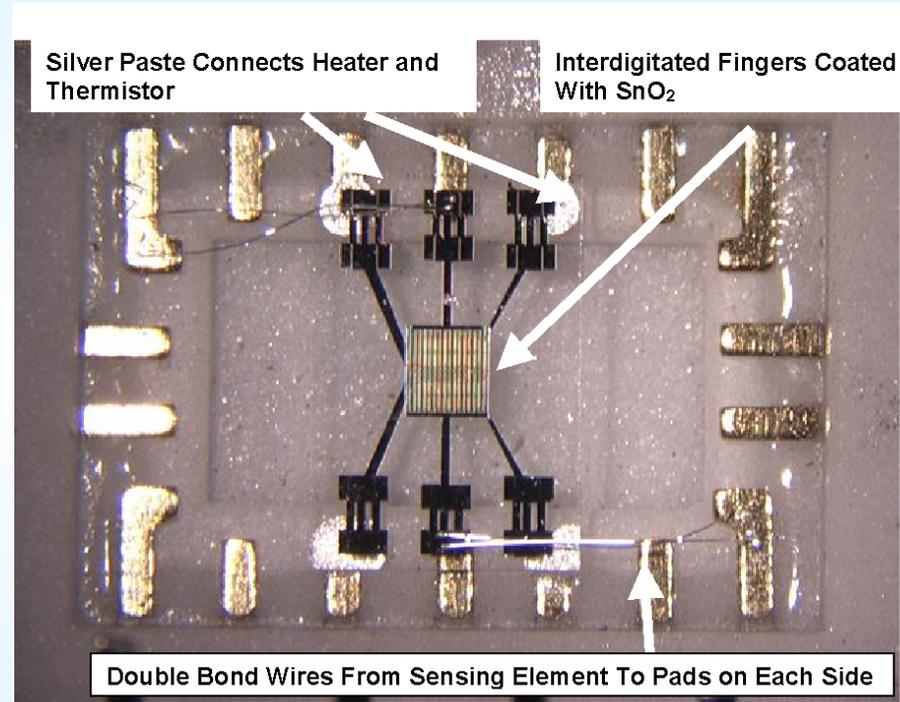
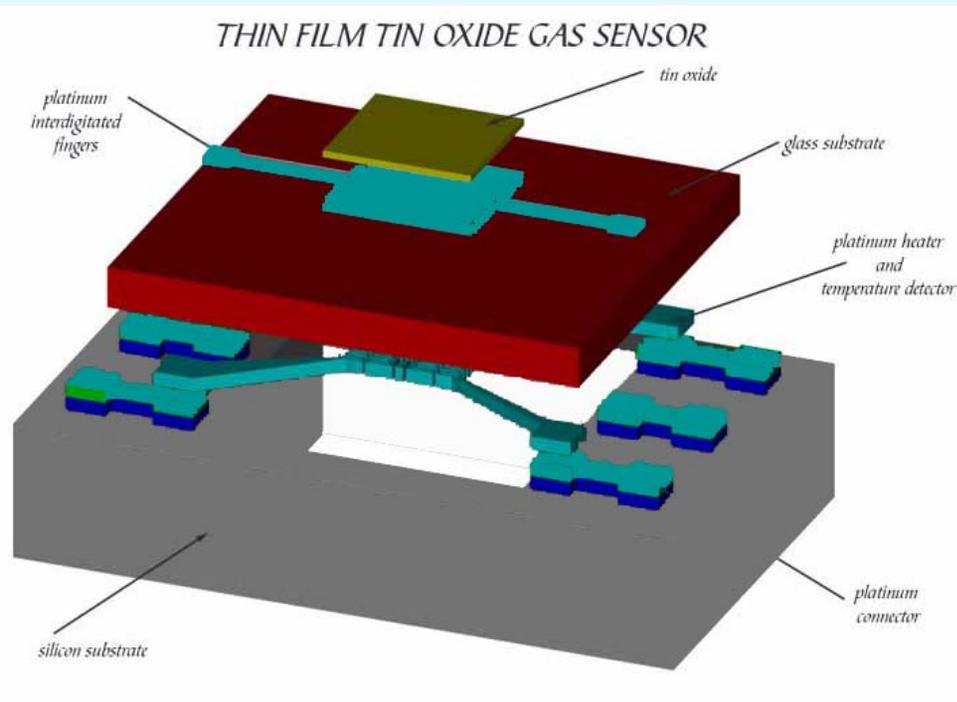


**Nanocrystalline SnO<sub>2</sub> after annealing at 600°C for 30 minutes.**

## Response of a Pt-doped Tin-oxide Sensor to a Range of CO Concentrations at 300°C in Air.



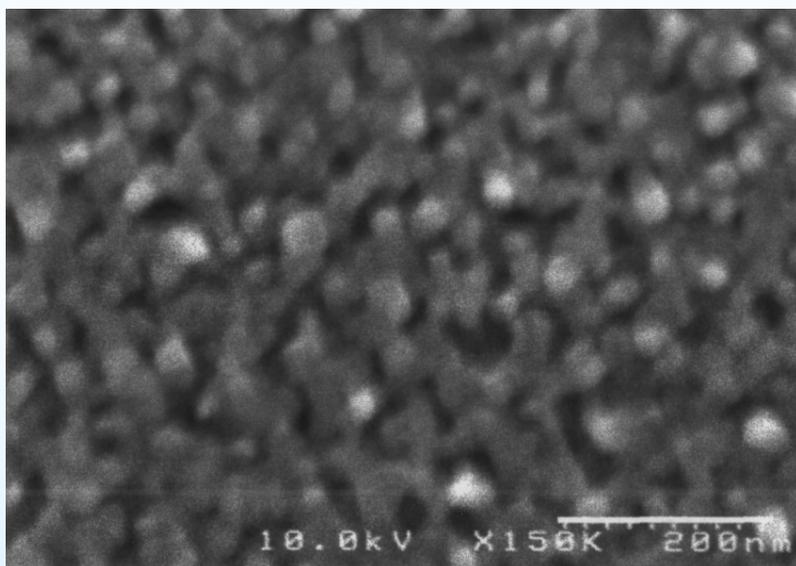
## STRUCTURE OF A $\text{SnO}_2$ BASED SENSOR ON A Si SUBSTRATE



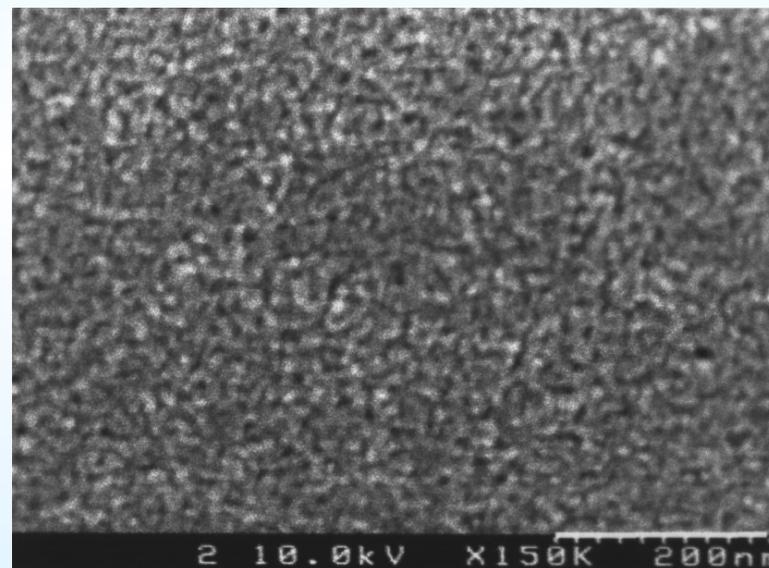
## EFFECT OF CALCINATION TIME AND SILICATE

700C calcined for 24 hours (on silicon substrate)

Undoped SnO<sub>2</sub> Film



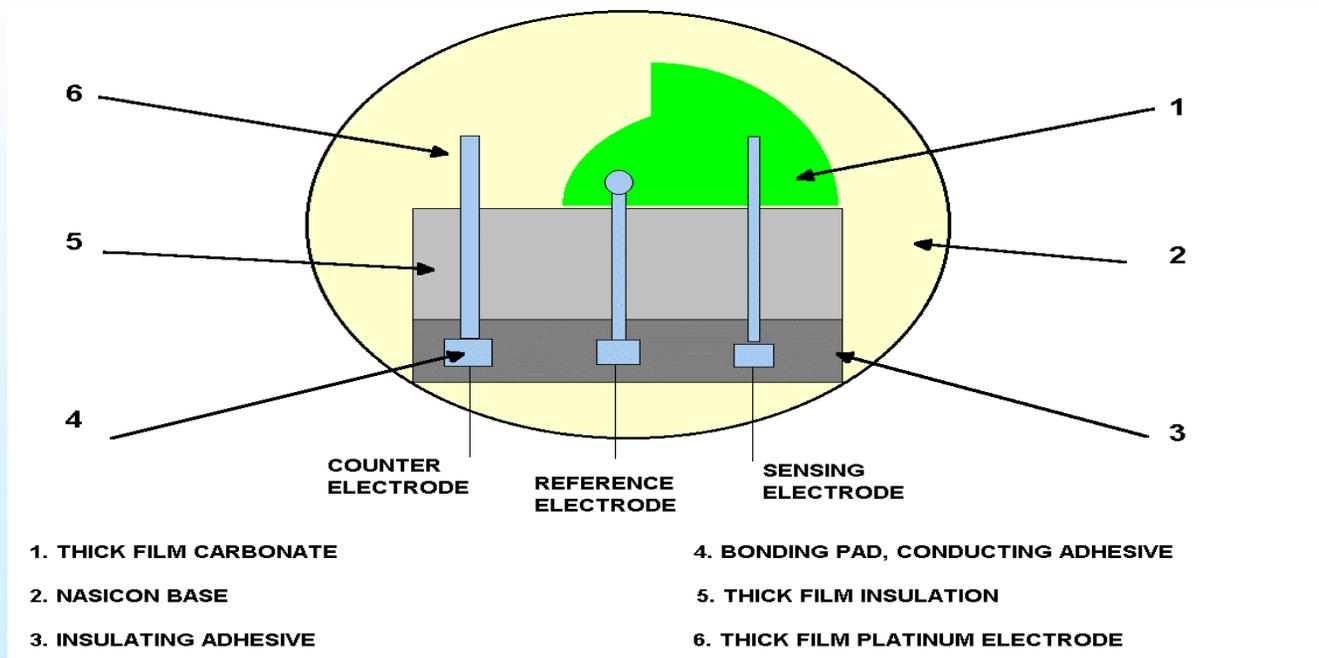
5 wt% SiO<sub>2</sub> doped SnO<sub>2</sub> Film



The particle and pore sizes increase with calcination time  
Silicate doping inhibits the particle growth

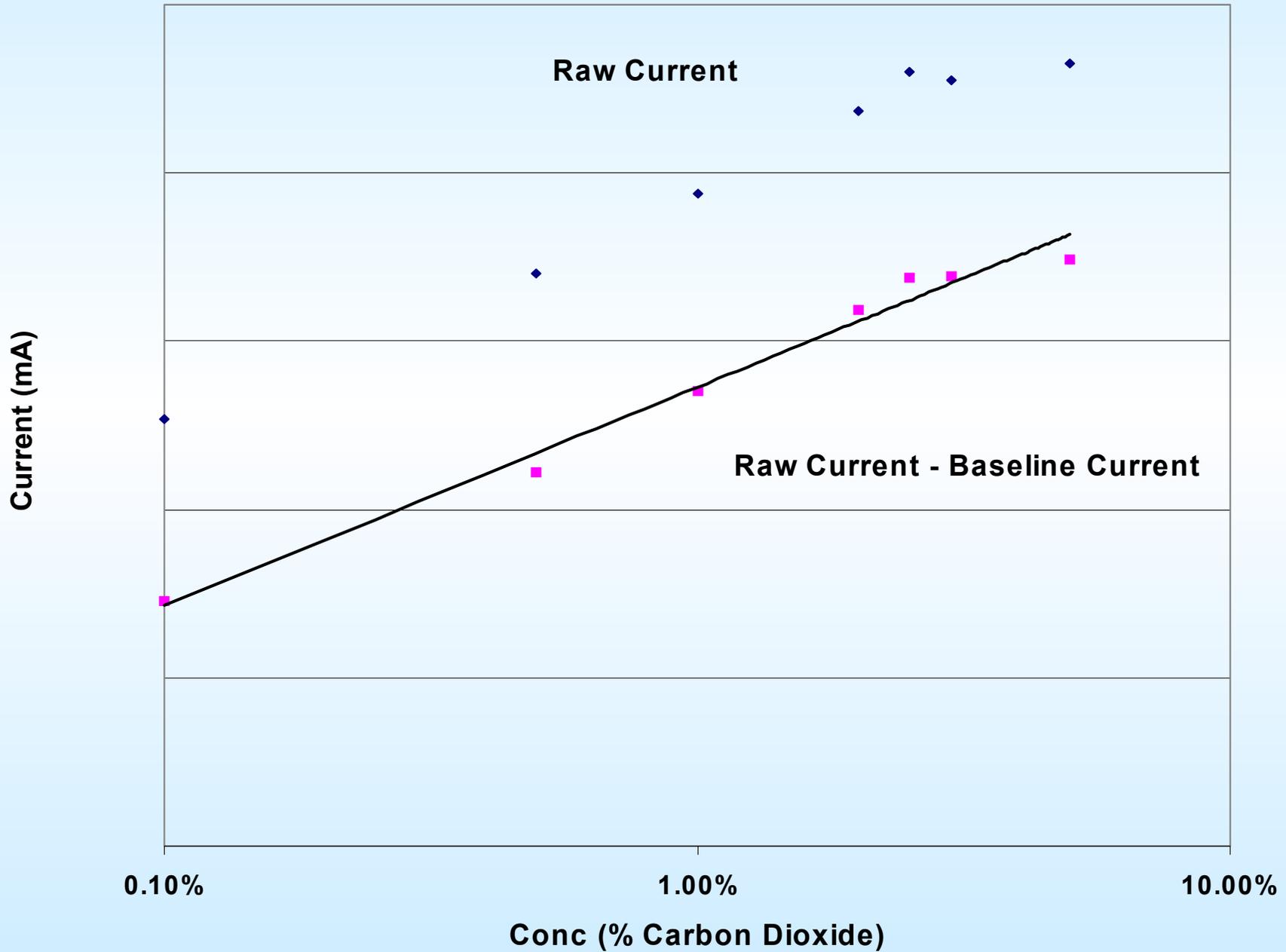
## *MICROFABRICATED NASICON BASED CO<sub>2</sub> SENSOR TECHNOLOGY*

- MICROFABRICATED FOR MINIMAL SIZE, WEIGHT AND POWER CONSUMPTION
- ELECTROCHEMICAL CELL DESIGN USING PROTON CONDUCTING NASICON AS ELECTROLYTE TO DETECT A RANGE OF CO<sub>2</sub> CONCENTRATIONS
- TEMPERATURE DETECTOR AND HEATER TO BE INCORPORATED INTO SENSOR STRUCTURE
- SENSOR TO BE COMBINED WITH CO SENSOR FOR SIMULTANEOUS CO/CO<sub>2</sub> DETECTION



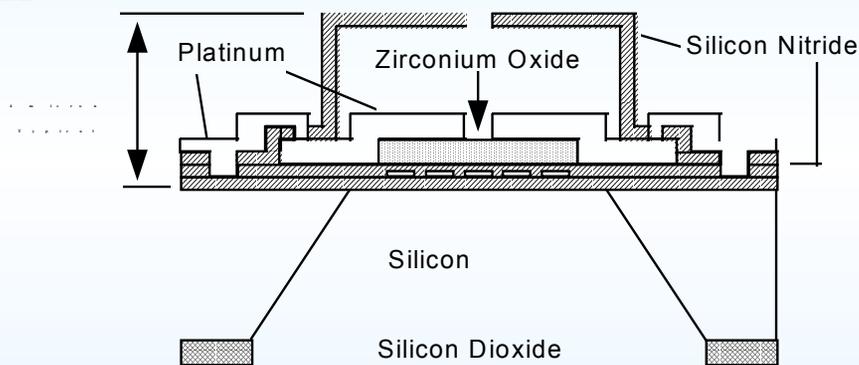
**STRUCTURE OF A NASICON-BASED ELECTROCHEMICAL CELL CO<sub>2</sub> SENSOR**

## CO2 Sensor Response- Peak Currents 3 min set delay - 2 min exposure delay

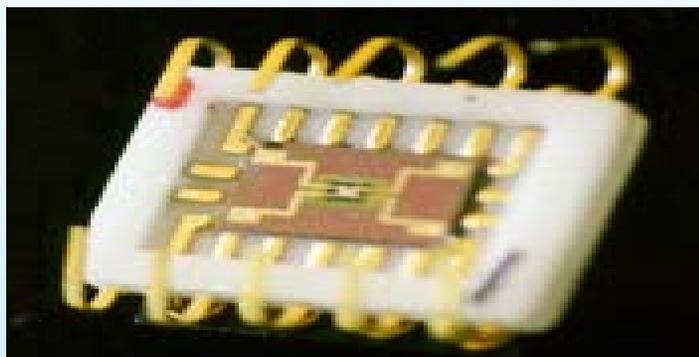


## MICROFABRICATED OXYGEN SENSOR TECHNOLOGY

- MICROFABRICATED AND MICROMACHINED FOR MINIMAL SIZE, WEIGHT AND POWER CONSUMPTION (LESS THAN 2 W FOR 600 C OPERATION)
- AMPEROMETRIC OPERATION ALLOWS MEASUREMENT OF OXYGEN OVER A WIDE CONCENTRATION RANGE (0-100%)
- CHAMBER STRUCTURE CONTROLS OXYGEN DIFFUSION RATE
- INCORPORATION OF OXYGEN SENSOR WITH OTHER SENSORS (E.G. HYDROGEN) IN THE SAME PACKAGE PLANNED



Not to scale:



ZrO<sub>2</sub> Oxygen Sensor

## SiC-BASED GAS SENSOR DEVELOPMENT

- THE USE OF SiC SEMICONDUCTORS ALLOWS SENSOR OPERATION AT TEMPERATURES WHICH ALLOW THE DETECTION OF HYDROCARBONS AND NO<sub>x</sub>
- SCHOTTKY DIODE DESIGN FOR HIGH SENSISTIVITY
- TEMPERATURE DETECTOR AND HEATER INCLUDED

OPERATION AT A RANGE OF TEMPERATURES

- WIDE RANGE OF APPLICATIONS

EMISSION MONITORING  
ENGINE HEALTH MONITORING  
ACTIVE COMBUSTION CONTROL  
HYDROCARBON FUEL LEAK DETECTION  
FIRE SAFETY

- PROTOTYPE SENSOR PACKAGE FABRICATED

- TWO APPROACHES

ALLOY ON SiC SUBSTRATE  
REACTIVE INSULATOR APPROACH

PACKAGED SiC-BASED SENSOR

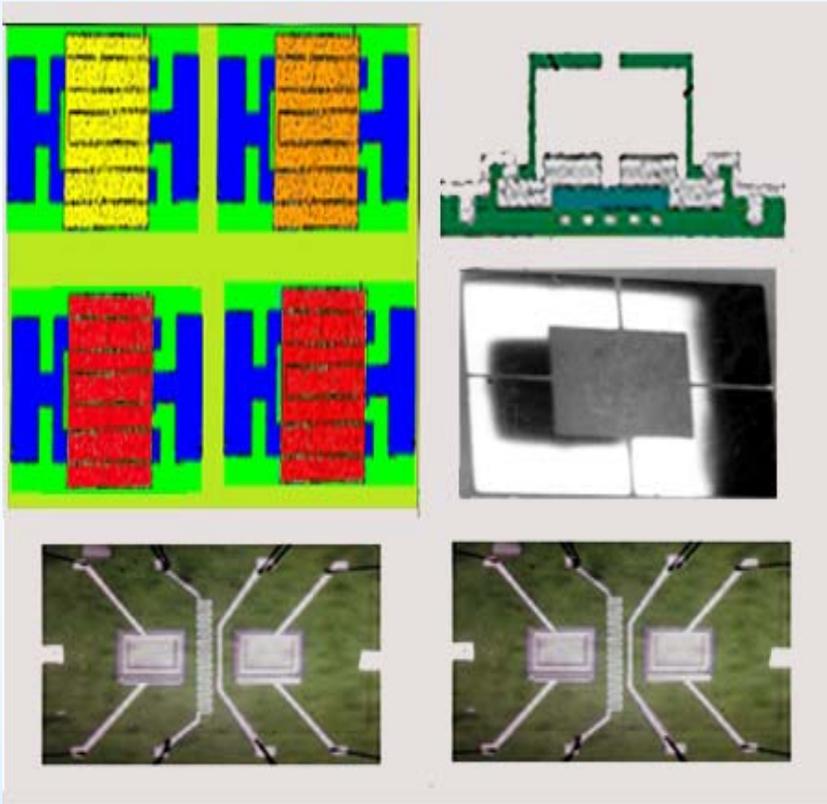


## HIGH TEMPERATURE GAS SENSOR ARRAY HIGH TEMPERATURE ELECTRONIC NOSE

SnO<sub>2</sub>  
Resistor

TiO<sub>2</sub>  
Resistor

Electrochemical Oxygen  
Sensor

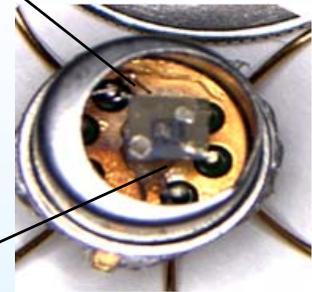


Selectively  
Filtered  
SnO<sub>2</sub> Resistors

SiC-Based  
Pressure Sensor

Metal-SiC  
Schottky diodes

Metal-Reactive Insulator  
SiC Schottky diodes

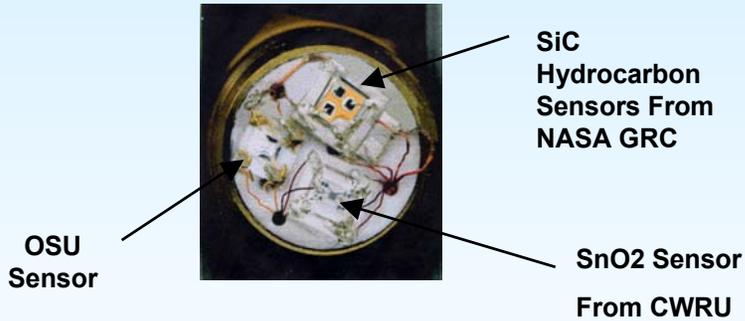




# Instrumentation & Control Technology Division

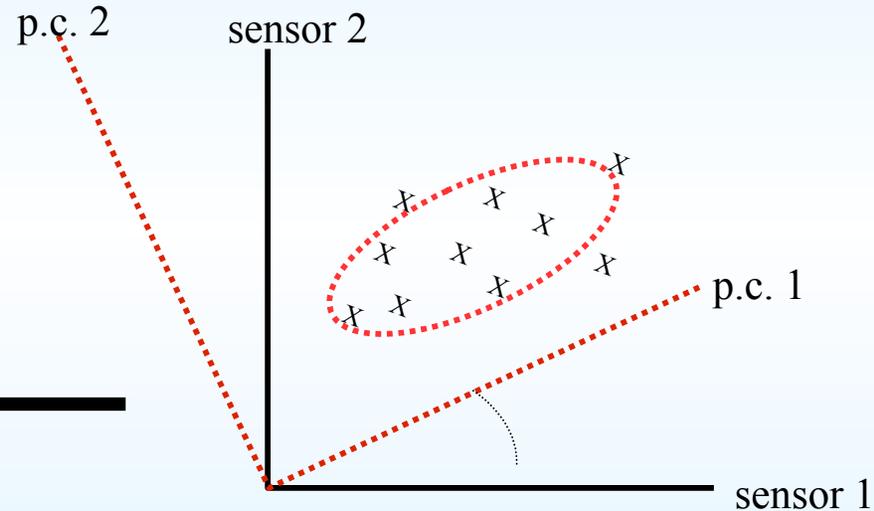
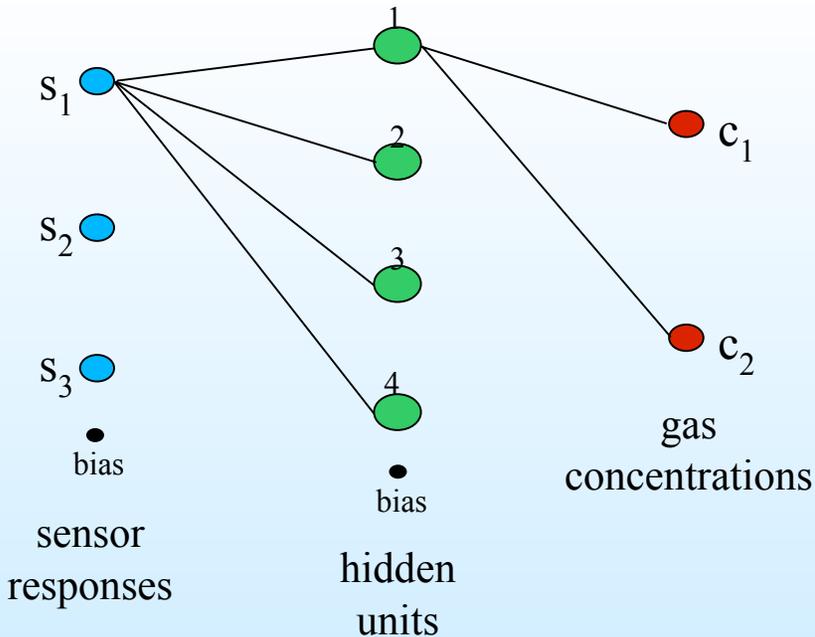
## SOFTWARE DEVELOPMENT TO INTERPRET SIGNALS PROTOTYPE SOFTWARE SYSTEM IMPLEMENTED

Prototype Sensor Head Used in Engine Test



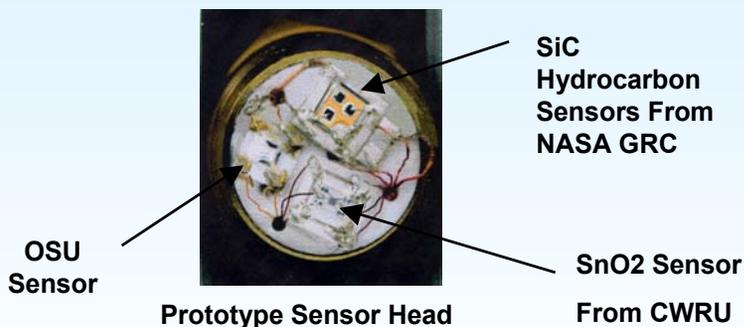
**BREAKING THE DATA SET DOWN INTO PRINCIPLE COMPONENTS HELPS DATA ANALYSIS. VERY DIFFERENT TYPES OF SENSORS AS USED IN THIS APPROACH IMPROVES THE CAPABILITIES OF THIS TECHNIQUE**

**GOAL: USE VERY DIFFERENT SENSORS IN AN ARRAY TO PROVIDE INFORMATION ON THE ENVIRONMENT**

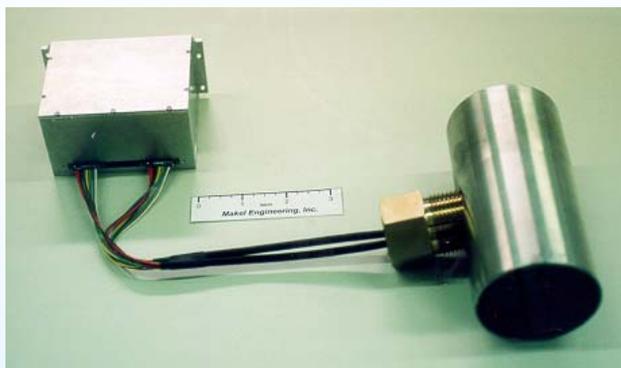


**FOR A GIVEN SET OF SENSOR RESPONSES, THE TRAINED NETWORK PREDICTS THE COMPONENT CONCENTRATIONS OF AN UNKNOWN MIXTURE OF, E.G. CO AND CH<sub>4</sub>**

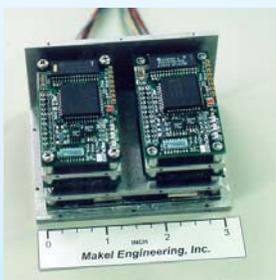
## *Brassboard Integrated Microsystem*



Sensor Operating Temperature 400 C  
with +/- 8 C stability in dynamic  
environment



Electronics and Sensor Head



### Sensors Tested

Oxygen (0 to 21%)

CO (0 to 3000 PPM)

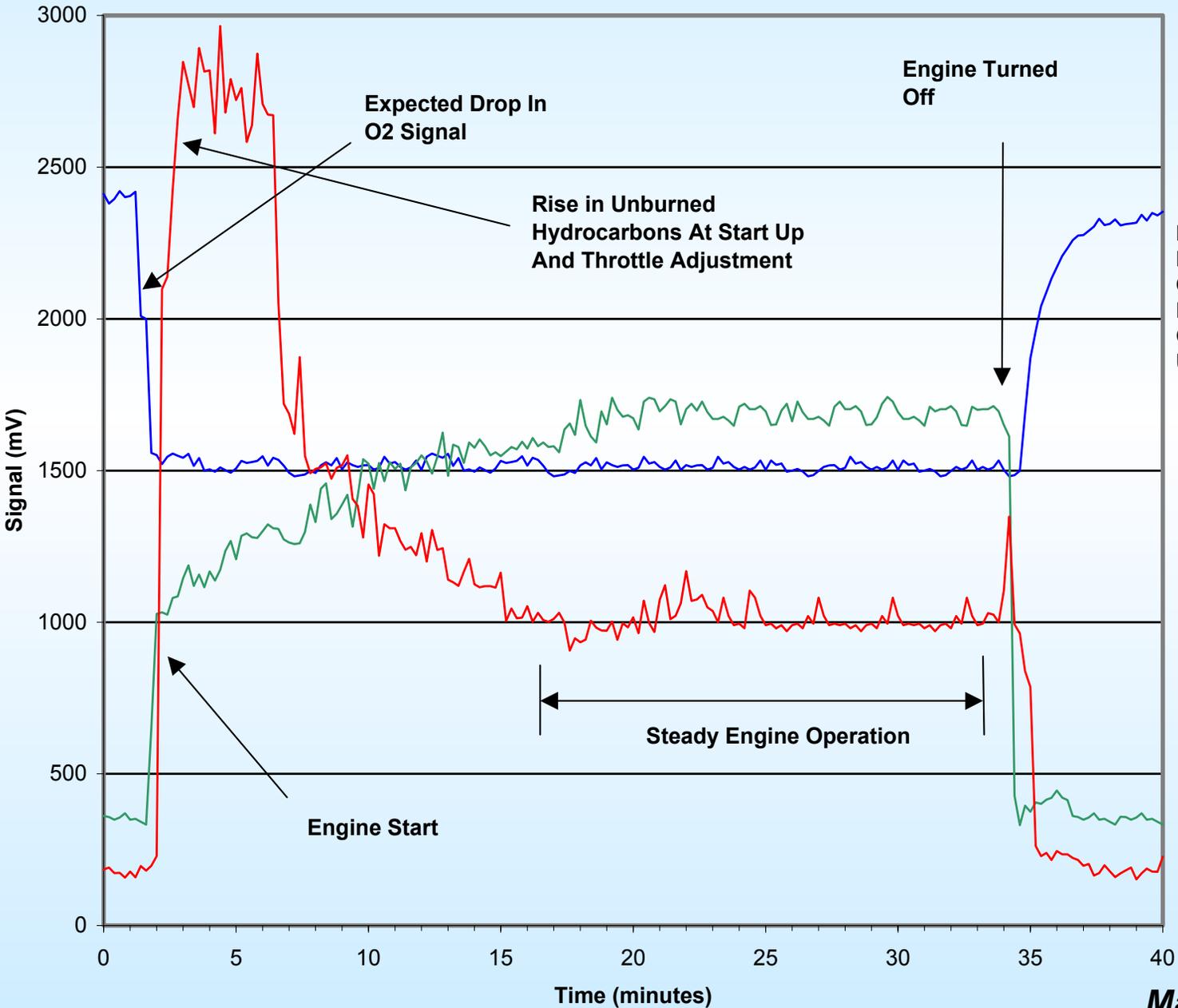
Hydrocarbons (0 to 2500 PPM C<sub>2</sub>H<sub>2</sub>)

NO<sub>x</sub> (0 - 300 PPM)



## Harsh Environment Demonstration Testing

1.9 liter, four cylinder HCII at U.C. Berkeley (propane/air)



Exhaust Gas Temperature = 337 °C  
Phi= 0.35  
O2=14%  
NOx<5 PPM  
CO =1400 PPM  
UHC =1200 PPM

- Oxygen Sensor
- SnO2 Sensor
- SiC Hydrocarbon Sensor





# **Instrumentation & Control Technology Division**

## **Micro-Fabricated Gas Sensors for Low False Alarms**

### **BACKGROUND**

- **COMMERCIAL PASSENGER AIRCRAFT TO HAVE CARGO SMOKE DETECTION & FIRE SUPPRESSION**
- **EXISTING SMOKE DETECTION TECHNOLOGY PRONE TO FALSE ALARMS (DIRT, DUST, MOISTURE, GASES, ETC.)**
  - **EMERGENCY DIVERSIONS & UNNECESSARY DISCHARGE OF HALON**
  - **FALSE ALARM RATE AS HIGH AS 200:1**
- **PRODUCE TECHNOLOGY WITH DIFFERENT FAILURE MECHANISMS**
  - **MONITOR CHEMICAL SPECIES**
  - **COMPLEMENTARY INFORMATION**
- **A NUMBER OF GASES EMITTED DURING FIRE/DEPENDENT ON FIRE**
  - **TWO GASES OF PARTICULAR INTEREST: CO/CO<sub>2</sub>**
- **IN COLLABORATION WITH:**
  - **FAA: TESTING**
  - **SANDIA NATIONAL LABS: MODELING**

## Micro-Fabricated Gas Sensors for Low False Alarms

### FEATURES

#### •MICROFABRICATED CO/CO<sub>2</sub> GAS SENSOR ARRAY

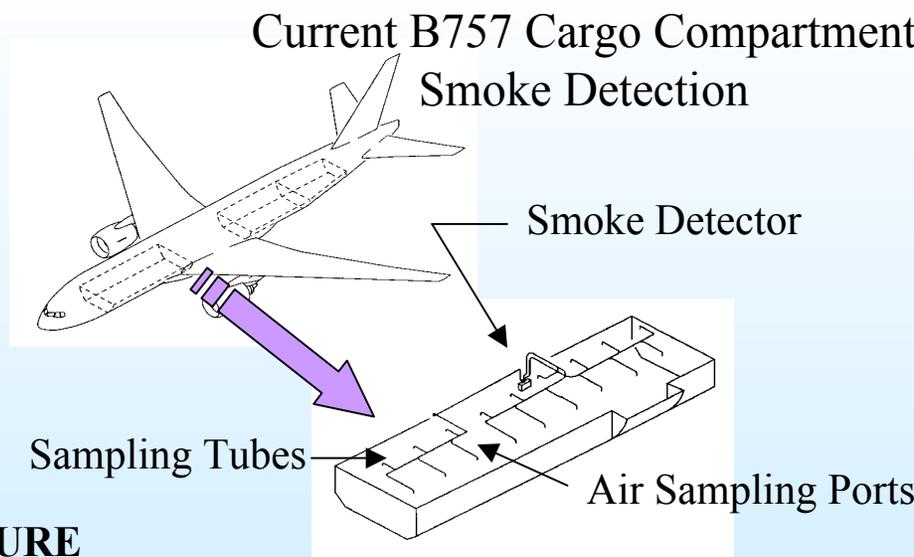
- CENTRAL TO APPROACH
- NANOCRYSTALLINE MATERIALS (IN CO SENSOR) PRODUCE MORE SENSITIVE, STABLE SENSORS
- TWO APPROACHES TO CO<sub>2</sub> DETECTION
- MINIMAL SIZE/WEIGHT/POWER

#### •CHEMICAL GAS SENSORS PROVIDE GASEOUS PRODUCT-OF-COMBUSTION INFORMATION

- SENSOR ARRAY CAN DETECT RANGE OF GAS SPECIES
- TO BE COMBINED WITH INTELLIGENT SOFTWARE FOR PATTERN RECOGNITION

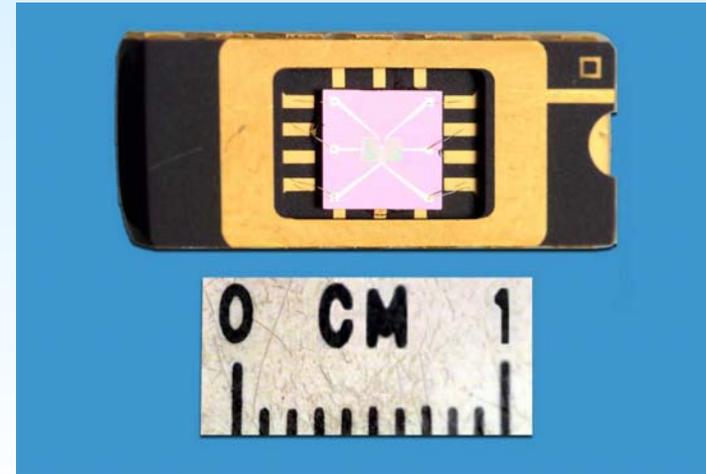
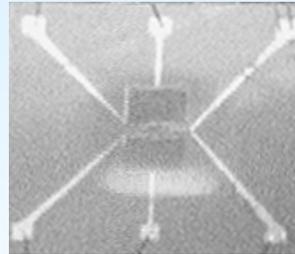
#### •BENEFITS

- DISCRIMINATE FIRES FROM NON-FIRES
- POTENTIAL SPIN-OFF TO HIGH-TEMPERATURE ENGINE MULTI SPECIES EMISSION CONTROL



## CHEMICAL SENSOR FIRE DETECTION

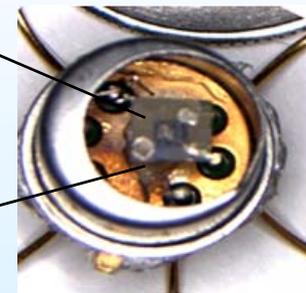
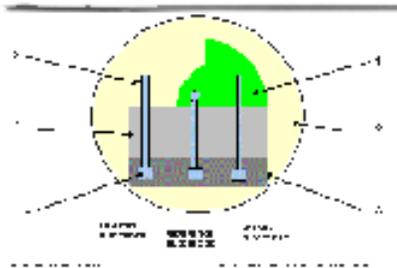
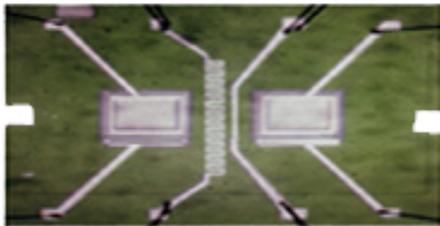
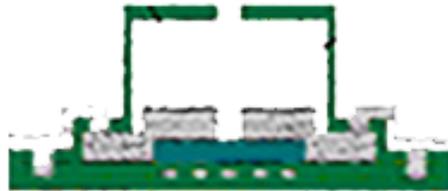
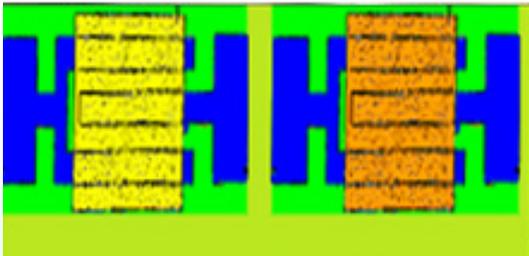
CO/CO2 SENSOR ON A CHIP



SnO2 Resistor (CO)

SnO2 Resistor (CO2)

Electrochemical Oxygen Sensors (O2 and Humidity)



Metal-SiC Schottky diodes

NASICON Based Electrochemical Cell



# **Instrumentation & Control Technology Division**

## **SUMMARY (CONT)**

- **AEROSPACE APPLICATIONS REQUIRE A RANGE OF CHEMICAL SENSING TECHNOLOGIES**
  
- **NEW FAMILY OF GAS SENSOR TECHNOLOGY BEING DEVELOPED TO MEET THESE NEEDS USING:**
  - **MICROFABRICATION AND MIRCROMACHING TECHNOLOGY**
  
  - **NANOMATERIALS**
  
  - **SiC-BASED SEMICONDUCTOR TECHNOLOGY**
  
- **FIRE DETECTION APPLICATIONS:**
  - **LEVERAGE EXISTING SENSOR DEVELOPMENT**
    - MULTIPLE SENSOR TYPES**
    - SENSOR ARRAYS**
    - ELECTRONICS**
  
  - **MINIATURIZED TECHNOLOGY WITH MINIMAL SIZE, WEIGHT, AND POWER CONSUMPTION**
  
  - **MULTIPLE POINT LOCATION**



## SUMMARY

- **COMBINATION OF SMOKE AND CHEMICAL SENSING TECHNOLOGY  
COMPLEMENTARY INFORMATION  
DIFFERING FAILURE MECHANISMS**
- **MULTIFUNCTIONAL SENSOR ARRAY  
CO/C02/HUMIDITY/HYDROCARBONS  
INTEGRATED SYSTEM  
SENSORS/POWER/COMMUNICATION**
- **FIRST GENERATION SYSTEM TO BE TESTED AT FAA IN FALL 01**
- **SANDIA MODELING: SENSOR PLACEMENT/INTERPRETATION OF RESULTS**
- **SYNERGY BETWEEN NASA SPACE AND AERONAUTICS APPLICATIONS**
- **LONG-TERM: INTEGRATE WITH MEMS-BASED SMOKE DETECTION SYSTEM FOR SPACE APPLICATIONS**